

CHARACTERISATION OF *MELASTOMA MALABATHRICUM* LEAVES AND  
CELLULOSE FOR REMOVAL OF METHYLENE BLUE IN SIMULATED  
WASTEWATER

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## ABSTRACT

Colors are an important class of pollutants, and disposal of colors in precious water resources should be prohibited. The regular commercial adsorbent is expensive, so it leads to researches on alternative low-cost adsorbents (LCAs) for such application. In this study, the usage of *Melastoma Malabathricum* cellulose as an adsorbent is discussed. The aim of this work is to extract cellulose from *Melastoma Malabathricum* (senduduk) leaves by alkali and bleaching treatment, characterising and, testing for colour removal of synthetic Methylene Blue colored wastewater. The characterisation techniques are initiated with chemical composition analysis before and after *Melastoma Malabathricum* cellulose extraction, results in percentage of cellulose increased to 90 % compared to raw leaves. The morphology of raw leaves and isolated cellulose are analysed by scanning electron microscopy (SEM) analysis. A structural analysis was carried out by Fourier transform infrared (FTIR) spectroscopy analysis and thermal stability was investigated by Thermogravimetric (TGA) analysis. The results indicated that the hemicelluloses and lignin were removed extensively from extracted cellulose. The thermal stability, purity and crystallinity of the cellulose were improved at various purification stages when compared to raw material. The adsorption method occupies a prominent place in color removal as it is effective and economical to application level. Thus, this technique is used to investigate the suitable condition for extracted cellulose of *Melastoma Malabathricum* by three parameters which are adsorbent dosage (g), initial color concentration (mg/L), and pH of Methylene blue colored wastewater used. Sample A is leaves (cellulose) which were soaked for 3 days/nights while Sample B is leaves (cellulose) soaked for 6 days/nights. By analyzing all the parameters, the color removal indicators prove senduduk cellulose B is the outstanding adsorbent to remove methylene blue efficiently compared to raw leaves and cellulose of sample A and B. The optimum pH for methylene removal by senduduk cellulose B is pH 7. About 69.63 mg/g adsorption capacity is achieved at optimum initial methylene blue concentration 20 mg/L, senduduk cellulose B dosage 0.10 g and pH 7. For percentage COD removal, 91 % reported at an optimum initial methylene blue concentration of 20 mg/L, senduduk cellulose B of dosage 0.10 g and pH 7. Therefore, soaked leaves for 6 days before chemical treatment and cellulose extraction results in better performance as adsorbent to remove color.

## ABSTRAK

Warna-warna adalah kelas penting bahan pencemar, dan pelupusan warna dalam sumber air yang berharga perlu dicegah. Penjerap komersial biasa adalah mahal, oleh itu ia membawa kepada kajian mengenai adsorben kos rendah alternatif (LCA) untuk penggunaan itu. Dalam kajian ini, penggunaan *Melastoma Malabathricum* selulosa dijadikan sebagai bahan penjerap yang dibincangkan. Tujuan kerja ini adalah untuk mengeluarkan selulosa dari *Melastoma malabathricum* (senduduk), menanggalkan untuk rawatan alkali dan pelunturan serta mencirikan bahan tersebut bagi mengujinya untuk penyingkiran warna sintetik Methylene Blue air sisa berwarna. Teknik-teknik pencirian dimulakan dengan penganalisan komposisi kimia sebelum dan selepas *Melastoma malabathricum* pengekstrakan selulosa, keputusan dalam peratusan daripada selulosa meningkat kepada 90% berbanding dengan daun mentah. Morfologi daun mentah dan selulosa terpencil dianalisis dengan pengimbasan electron microscopy (SEM). Penganalisan struktur dijalankan oleh Fourier transform infrared (FTIR) spectroscopy dan kestabilan haba yang disiasat oleh Thermogravimetric (TGA) analisis. Keputusan menunjukkan bahawa hemiselulosa dan lignin telah dikeluarkan secara meluas dari selulosa yang diekstrak. Ketahanan haba, kebersihan dan penghabluran sellulosa telah bertambah baik di pelbagai peringkat pembersihan jika dibandingkan dengan bahan mentah. Kaedah penjerapan menduduki tempat yang penting dalam penyingkiran warna kerana ia adalah berkesan ke tahap ekonomi. Oleh itu, teknik ini digunakan untuk menyiasat keadaan yang sesuai untuk selulosa yang diekstrak daripada *Melastoma malabathricum* oleh tiga parameter iaitu dos penjerap (g), kepekatan warna permulaan (mg / L), dan pH air sisa berwarna biru yang digunakan. Sampel A adalah daun (selulosa) yang direndam selama 3 hari atau malam manakala sampel B adalah daun (selulosa) yang direndam selama 6 hari / malam. Dengan menganalisis semua parameter, penunjuk penyingkiran warna membuktikan selulosa senduduk B adalah contoh baik untuk membuang methylene blue dengan cekap berbanding daun mentah dan selulosa sampel A dan B. pH optimum untuk penyingkiran methylene blue oleh selulosa senduduk B adalah pH 7. Penjerapan 69.63 mg/g dicapai pada kepekatan awal optimum methylene blue 20 mg/L, selulosa senduduk B dos 0.10 g dan pH 7. Untuk peratusan penyingkiran COD, 91% dilaporkan untuk kepekatan awal sebanyak optimum methylene blue 20 mg/L, selulosa senduduk B dos 0.10 g dan pH 7. Oleh itu, daun direndam selama 6 hari sebelum rawatan kimia dan perahan selulosa adalah lebih baik sebagai penjerap untuk membuang warna.

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**LIST OF SYMBOLS**

g	-	gram
H <sub>2</sub> O <sub>2</sub>	-	Hydrogen peroxide
l	-	litre
mg/L	-	milligram/litre
°C	-	degree Celsius
θ	-	Angle
λ	-	Wavelength

## LIST OF ABBREVIATION

BOD	-	Biological oxygen demand
COD	-	Chemical oxygen demand
FTIR	-	Fourier transform infrared
NaClO <sub>2</sub>	-	Sodium chlorite
NaOH	-	Sodium hydroxide
SEM	-	Scanning electron microscopy
TDS	-	Total dissolved solids
TGA	-	Thermogravimetric analysis
TSS	-	Total suspended solids
XRD	-	X-ray diffraction

## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 Research Background**

Looking at different known types of contamination, water contamination is the worst since water is the prime need of mankind and extremely fundamental for every single living thing to survive. Be that as it may, water contamination has turned into a general wonder that is more genuine in the creating nations to consider on release of for the most part untreated or incompletely treated city and mechanical wastewaters that damages amphibian living spaces (Sharma *et al.* 2007). Years of increased industrial, agricultural and domestic activities have results in release of huge amount of wastewater with high in composition of toxic pollutants (Bhatnagar *et al.*, 2006).

The availability of clean water is becoming very low and very challenging. Moreover, the demand for water (“Water for People Water for Life” United Nations World Water Development Report UNESCO) has risen for the sectors of industrial, agricultural and domestic which consuming 70%, 22% and 8% of the available clean water respectively. It has been one of the challenging tasks for the researchers and practitioners to meet the clean water demand worldwide. As the awareness of pollutants present in water affect all living things, pollution control and management is highly advised and prioritised in pollution prone areas.

One of the important classes of pollutants is color. Colors have synthetic origin and difficult to treat as colors are made of complex molecular structure that makes them more rigid, unbreakable and non-biodegradable (Forgacs *et al.* 2004). Colors adsorb sunlight which affects the intensity of light absorbed by hydrophytes and phytoplankton and slows down photosynthesis and dissolved oxygen in the aquatic and it eventually results in high chemical oxygen demand (COD) (Rangabhashiyam *et al.* 2013). Colored effluent made of harmful organic and inorganic chemicals that exhibit toxic and carcinogenic effects toward biological environment. Industries like textile, dyestuffs, paper, and plastics use color for their production and generate colored wastewater (Forgacs *et al.* 2004).

According to Monika *et al.* (2012), color is the initial and first contaminant to be found and identified in the wastewater. Pearce *et al.* (2003) stated that more than 100,000 types of color are available commercially and over  $7 \times 10^5$  tonnes of color are generated annually. Although, the exact data on amount of color discharged in the environment is not available, it is estimated that a loss of 1-2% in production. Even though, there are many growing environmental protection towards to the industrial development induces eco-friendly technologies, the discharge of synthetic color to the environment results serious challenge to the researchers. Comparing different techniques of color removal from wastewater, it is proven that adsorption technique is one of the best technology and presented good results in the removal of different types of coloring materials (Jain, 2003). Adsorption procedures to expel colors from wastewater have been generally utilized. It has been observed to be a prudent and powerful treatment technique for expulsion of colors because of its sludge free clean operation (Kushwaha, *et al.*, 2014).

## 1.2 Problem Statement

Melastoma Malabathricum (Senduduk) is a plant use in traditional Malay society prescription for treatment of diarrhea, dysentery, toothache, fart and hemorrhoids. Anthocyanins are an important group of water-dissolvable plant pigments normally

found in different products of the soil. Two major anthocyanins aglycon in senduduk are cyanidin-3-glucoside and cyanidin-3, 5-diglucoside (Aishah *et. al.*, 2013). These anthocyanins are important pigments which used as natural food colorant to replace synthetic dyes. Thus, it is essential to remove the hemicellulose and lignin from senduduk leaves and use senduduk cellulose in color removing in wastewater in this research. Experiments using raw senduduk leaves will act as control experiment.

According to Hugh *et. al.* (2009) from National University of Singapore who has published a research on the potential of native woody plants for enhancing the urban waterways, herbaceous (non-woody) plants are hyper accumulators compared to woody plants. The higher uptake rate of herbaceous increases removed pollutants in wastewater (Pulford and Watson, 2005). Native senduduk plant is the best example for phytoextraction which has been applied by Hugh *et. al.* (2009) to enhance Singapore waterways which consists of domestic and industrial wastewater.

Discharge of wastewater containing color mixes into water sources will deplete dissolved oxygen content in water furthermore repress daylight from reaching to the water sources. A portion of the color mixes can discharge poisonous mixes making deterioration biological system and human wellbeing. Release of color-bearing wastewater into common streams and waterways from textile, paper, rug, leather, refinery, and printing commercial ventures postures extreme issues since colors grant harmfulness to the oceanic life and cause harm to the stylish way of nature. The color containing wastewater is normally discharged straightforwardly into the adjacent channels, rivers, stagnant, lakes or tidal ponds. The discharge of colors into wastewaters by different industries postures genuine ecological issues because of different colors' persistent and obstinate nature.

Wastewater containing colors are extremely hard to treat, since the colors are recalcitrant natural particles, impervious to biological degradation and are steady to light. The waste materials from the textile wastewater consists of large portion of biochemical oxygen demand (BOD), chemical oxygen demand (COD), pH, temperature, turbidity, toxic chemicals, high concentrations of heavy metal and total dissolved solids (Sharma *et al.* 2007; Garg *et. al.*, 2004). Textile wastewater is a mixture of colorants (colors and

pigments) and organic compounds used as cleaning agents. Different types of color are discharged from textile industries in terms of molecular weight, structures and biodegradability (Pala and Tokat, 2002). Due to discharge from textile industry that results in decline of algae growth, the water body leads to devoid of fauna (Sharma *et al.*, 2007). Therefore, it is clearly understood that textile wastewater are highly toxic to the environment.

As color in wastewater cannot be efficiently removed by traditional methods because of the chemical stability of these pollutants, the adsorption of color on inexpensive and efficient solid supports is considered as a simple and natural technique for color removal from textile wastewater. Adsorption is the superior process for water purification applications. Adsorption has been observed to be better than different procedures for water reuse regarding introductory cost, adaptability and simplicity of design and ease of operation as review (Ashoka and Inamdar, 2010; Tsai *et.al.*, 2001, Kannan *et. al.*, 2010). The variety of adsorption in inorganic and organic matrices has been measured and their capacity to remove color has been studied in previous paper (Forgacs *et al*, 2004). Ho and McKay (1998) also states that, adsorption is one of the treatment method, gives the finest results in removing different coloring materials. This is because adsorption removes the entire color molecule without leaving fragments in the effluent.

A wide range of adsorbents have been extensively used in effluent treatment. Among many new technologies, utilizing activated carbon for color removal from effluent is prominent technology (Kadirvelu *et. al.* 2003). However, using adsorbents in large scale is quite expensive for removal of color. Thus, many researchers are studying the application of natural adsorbent as an alternative to costly adsorbents (Ponnusami *et. al.* 2008).

A specialist examines the adsorption of Congo red on carbon prepared from neem leaf litter and raw neem Leaf. As indicated by the authors, adsorption of Congo red on Neem leaf litter demonstrated the most noteworthy adsorption limits contrasted with some other adsorbents (Manikanda *et. al.*, 2012). It was watched that the adsorbent is powerful for the removal of anionic colors in a wastewater treatment process. (Chiou *et.*



*al.*, 2004; Liew *et. al.*, 2005) study initiated carbons prepared from teak leaf, maize corn and babool tree husk were utilized to study adsorption of red industrial color under different test conditions. The authors exhibited that the adsorbents were powerful for the removal of Methylene red industrial color. Among the coloring agents, methylene blue (MB) is a standout amongst the most broadly utilized colors, particularly in textile industry (Sajab *et. al.*, 2011).

### **1.3 Objectives**

The target of this work is to explore the capability of adsorbent (cellulose) obtained from low cost local accessible senduduk leaves for the removal Methylene Blue color. The research is to achieve the following objectives:

- To extract cellulose from senduduk leaves
- To characterize senduduk leaves and its cellulose in terms of chemical composition, Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR) and Thermogravimetric (TGA) analysis
- To investigate the suitable operating conditions for extracted cellulose of senduduk leaves for Methylene blue color removal of wastewater

### **1.4 Scope**

In order to achieve the objectives, certain scopes have been set which controls the range of the research.

1. The study is firstly; prioritize on extracting cellulose from senduduk leaves. Chemical treatment (alkali treatment and bleaching respectively) has been applied on raw senduduk leaves in the cellulose extracting process (Fortunati *et al.*, 2012). Extraction method is used to characterize of the adsorbent from plant fibers by investigating the origin of the fibers (Sheltami *et al.* 2012).
2. Secondly, the cellulose extracted from senduduk leaves is characterized by chemical composition, morphology (SEM analysis), functional group (FTIR spectroscopy analysis) and thermal stability (TGA analysis) with reference of senduduk leaves (blank) (Fortunati E. *et al.*, 2012; Southon J. R., 2010). The characterisation methods are repeated for raw senduduk leaves and senduduk cellulose (after alkali treatment and after bleaching treatment).
3. Thirdly, the performance of the extracted cellulose as adsorbent is tested using adsorption process using batch adsorption experiments. The study also researched parameters that might influence the color adsorption, including initial color concentration (mg/L), pH of the wastewater are used and adsorbent dosage (g). The treated wastewater was analyzed for percentage reduction of initial color concentration and chemical oxygen demand (COD).
4. The range for pH of 3 – 11 is chosen based on preliminary studies by Weng *et al* (2009), Bhatnagar *et. al.* (2010) and Mohan *et al.* (2002) using herbaceous leaves. Immich *et al.*, 2009 is the reference for initial color concentration (mg/L) for removal of Remazol Blue RR using neem leaves, initial color concentration below 20 mg/L was used in the study. The range of adsorbent dosage of 0.02 – 0.10 g is used in this study as per referred to Pandhare *et.al.* (2013), an experiment done in color removal by Neem Leaves Powder from methyl red solution.

## **1.5 Significance of Study**

The results of this study will be helpful for utilizing this common waste either senduduk leaves or senduduk cellulose as a monetary nature based bio-adsorbent in the removal of methylene blue from wastewater. This helps to protect the waters sources of sea, lake or river from wastewater through harmful color effluent. It can be essential to realize the importance of balancing the environment, social, economic viability towards sustainable development.

The generation of cellulose from this underutilized agro-waste has business application potential that can increase the value of the senduduk development, produce additional wage for ranchers furthermore offer assistance in agribusiness expansion. What's more, the use of these natural materials permits a huge lessening both in the volume of waste amassed in the earth, as in the extraction of raw materials.

Besides that, the research provides opportunity for the researchers to make innovative adsorbent production to meet the high demand as well as low cost. The data obtained from this study would be useful and could be further verified for betterment of extraction of cellulose from senduduk leaves as environmental friendly and low cost adsorbent to be used in wastewater to remove color.

## **1.6 Thesis Outline**

There are five chapters in this thesis and each chapter describes the sequences of the research. Chapter 1 briefs the research of color removal of textile wastewater by adsorption. This chapter presents the problem statement, research objectives, and scopes of the study and the significance of the study. Chapter 2 consists of the literature review of related knowledge about wastewater, color and the available treatments to remove color in wastewater. Besides, this chapter also covers the previous studies of characterization of the adsorbent. Chapter 3 explains the materials and methods being used in the study. The chapter describes the experimental procedure for the extraction of

cellulose from senduduk leaves and the adsorption preparation and process to treat methylene blue color. Chapter 4 presents the results and discussion from the study which compresses extraction of cellulose from senduduk leaves, characterization of the adsorbent and performance of the adsorption to treat methylene blue color. Chapter 5 refers to overall conclusions that are based on the findings obtained in the results and discussion which is explained in Chapter 4. Furthermore, recommendations for future research are also added in this chapter to enhance the structure of study as well as the results.

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